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# PROPERTIES OF A TZP/ $\text{Al}_2\text{O}_3$ COMPOSITE AFTER LONG-TERM EXPOSURE AT 1000°C

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#### ABSTRACT

A commercially available tetragonal zirconia polycrystal ceramic containing 20 w/o  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> was examined for potential use in advanced engines. Room temperature strength and toughness were determined, as well as the static fatigue life between 800°C and 1200°C. All of these properties were determined before and after a 100 hour exposure at 1000°C. The room temperature flexure strength was 1838 MPa before exposure and it decreased 23% after exposure. The toughness was  $\approx 5$  MPa $\sqrt{m}$  and did not change with the exposure. Static fatigue testing revealed that fracture below 1100°C was due to the cracks created during machining. At 1100°C and higher slow crack growth became the dominant failure mechanism. Overall, the high temperature static fatigue life is better than yttria-tetragonal zirconia polycrystal materials without Al<sub>2</sub>O<sub>3</sub> but it still does not meet the strength-at-temperature requirements ( $>800$  MPa at 982°C) for use in advanced engines.

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## **FOREWORD**

The work described in this report was supported by the U.S. Army Materiel Command under the D650 Program: Exploitation of Foreign Technology, Project No. 89148. The project has two objectives:

- To evaluate a commercially available ceramic composite, which consists of tetragonal zirconia ( $\text{ZrO}_2$ ) polycrystals (TZP), and alumina ( $\text{Al}_2\text{O}_3$ ), for potential use in the next generation of advanced heat engines.
- To examine the phenomenon of superplasticity which occurs in this material.

This report focuses on the first objective.

## INTRODUCTION

Previous work<sup>1</sup> has shown that tetragonal zirconia polycrystal materials, partially stabilized with yttria ( $Y_2O_3$ ), (Y-TZPs) can have toughness and strength values as high as 7 MPa $\sqrt{m}$  and 1100 MPa, respectively. The excellent toughness is due to the stress-assisted transformation of the metastable tetragonal (t) phase to the stable monoclinic (m) phase. The high strength is due to a combination of the fine grain size of the material and the transformation.

Despite having these excellent properties Y-TZP materials are not being used for structural applications due to a significant decrease in properties after long-term exposure to elevated temperatures,<sup>1-3</sup> at elevated temperatures under stress,<sup>2,4</sup> and after exposure to 200°C to 300°C in the presence of water vapor.<sup>5-8</sup> In all three cases the loss of toughness and strength is related to a decrease in the amount of t-ZrO<sub>2</sub> present. The mechanisms which cause the reduction in t-ZrO<sub>2</sub> vary based upon the environmental conditions and are discussed in detail in the reference papers.

During the past five years studies at TOSOH Corporation (formerly Toyo Soda Manufacturing Company) have shown that the addition of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> to a Y-TZP can significantly improve the strength but with a loss in toughness.<sup>9-11</sup> It was found that a maximum strength of 2400 MPa (determined from three-point flexure) and a toughness of 6 MPa $\sqrt{m}$  could be achieved when 20 to 30 vol%  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> was added to the Y-TZP.<sup>9</sup>

Lange<sup>12</sup> reported that there are several factors which affect the transformability of the tetragonal zirconia grains: the stabilizer content, the grain size, and the matrix forces which constrain the grains. In this composite it is believed that the addition of the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> enhances the rigidity of the matrix (MOE<sub>Al<sub>2</sub>O<sub>3</sub></sub>  $\approx$  406 GPa versus MOE<sub>Y-TZP</sub>  $\approx$  210 GPa). The enhanced rigidity increases the critical stress necessary to initiate the tetragonal-to-monoclinic transformation, thus increasing the overall strength. The reduction in toughness is simply due to the decrease in the volume fraction of t-ZrO<sub>2</sub>.<sup>10,11</sup>

This report summarizes a study to examine the effects of long-term exposure at 1000°C on the room and elevated temperature properties of a Y-TZP containing 20 w/o  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>.

## MATERIAL

The material evaluated in this study was produced by TOSOH Corporation of Tokyo, Japan using TOSOH TZ-3Y20A "Super Z" zirconia/alumina powder. The powder was formed into billets 6" x 6" x 0.5" in size through a sinter/hot isostatic press (HIP) process. Primary sintering was done at  $\approx$ 1400°C followed by HIPing at  $\approx$ 1500°C. The composition of the powder, according to company literature, is 75.7 w/o tetragonal zirconia (partially stabilized by 4.2 w/o yttria) with 20 w/o  $\alpha$ -alumina. Typical impurity content is as follows: SiO<sub>2</sub> (0.01 w/o), Fe<sub>2</sub>O<sub>3</sub> (0.005 w/o), and Na<sub>2</sub>O (0.007 w/o). Pertinent properties reported by the manufacturer are listed in Table 1.

Table 1. MANUFACTURERS ROOM TEMPERATURE  
PROPERTY VALUES

Density (g/cc)	5.5
Young's Modulus (GPa)	260
Flexure Strength* (MPa)	2400
Fracture Toughness† (MPa*√m)	6
Hardness (kg/mm <sup>2</sup> )	1470

\*Three-point flexure strength (specimen size 3 mm x  
4 mm x 40 mm, test span 30 mm)

†Microindentation method

### EXPERIMENTAL PROCEDURE

Billets of the "Super Z" material were obtained and machined into type "B" flexure bars (3 mm x 4 mm x 50 mm) according to MIL-STD-1942A. The bulk density of each bar was determined by measuring the mass and geometry, and a pulse-echo ultrasonic technique was used to determine the modulus of elasticity (MOE). Half of the bars were then heat treated for 100 hours at 1000°C. Heat treatments were done in air, at laboratory ambient humidity, in an unstressed condition on silicon carbide knife edges for uniform thermal treatment. The knife edges supported the bars well outside the test area to assure that any reactions would have no effect on the property evaluation. The density and MOE were again measured after the heat treatment. Strength, toughness, and the static fatigue behavior at elevated temperatures were determined before and after the heat treatment.

Room temperature strength was determined using four-point flexure tests, as outlined by MIL-STD-1942A, with inner and outer spans of 20 mm and 40 mm, respectively, and a cross-head speed of 0.5 mm/min. The mean strength was determined based upon a minimum of five bars from each condition. The fracture surfaces of each bar were examined optically and selected bars were examined with a scanning electron microscope (SEM) in an attempt to characterize the strength-limiting flaw(s). Fractographic analysis was carried out according to Reference 13.

The "effective" fracture toughness ( $K_{IC}$ ) was measured by subjecting five bars per condition to the Vickers indentation-strength technique outlined in Reference 14 using a 10 kg indentation load. Prior to indentation, one 4 mm x 50 mm face of each bar was polished to a finish of 2-microinch RMS or better. This was done to remove any existing machining damage. The bars were broken in four-point flexure immediately after indentation to minimize any environmental effects. Each bar was then examined to be sure that the fracture did occur at the indent.

Stepped-temperature stress-rupture (STSR) testing was used to analyze the static fatigue life of the material between 800°C and 1200°C. STSR testing was done following the procedure outlined by Quinn and Katz.<sup>15</sup> This type of test allows for rapid screening of the materials static fatigue life over a wide range of temperatures while only using a small number of bars.

The linear intercept method<sup>16</sup> was used to determine the average grain size. This method was applied to SEM micrographs of specimens which were thermally etched at 1650°C for 15 minutes.



The type and amount of zirconia phases present were determined using X-ray diffraction with  $\text{CuK}\alpha$  radiation over an angular range of  $25^\circ$  to  $40^\circ$   $2\theta$ . The surface fraction of monoclinic zirconia and the fraction of tetragonal-plus-cubic zirconia was calculated using the equations and constants from Reference 2 which take into account the difficulty in deconvoluting the tetragonal (101) and cubic (111) peaks.

## RESULTS AND DISCUSSION

### Room Temperature Properties

The room temperature properties, before and after heat treatment, are summarized in Table 2. All properties listed, with the exception of the flexure strength, are in excellent agreement with the values reported by the manufacturer and do not change after the long-term exposure.

Table 2. PROPERTY VALUES DETERMINED IN THIS STUDY

Property	AR*	AHT†
Density (g/cc)	5.5	5.5
Young's Modulus (GPa)	258	259
Mean Flexure Strength (MPa)	1838	1493
Standard Deviation (MPa)	308	59
Weibull Modulus	6	24
Fracture Toughness ( $\text{MPa}\sqrt{\text{m}}$ )	5	5
Knoop Hardness (300 g load) ( $\text{kg}/\text{mm}^2$ )	1476	1483

\*AR = As-received

†AHT = After Heat Treatment

The strength discrepancy between the manufacturer's value and as-received value obtained in this study is  $\approx 23\%$ . This can be accounted for by the different test methods and specimen sizes that were used. Weil and Daniel<sup>17</sup> provided a statistical method, based upon the Weibull distribution, of comparing strength values obtained from different test methods and/or specimen sizes.

Assuming the material follows this distribution, the effect of specimen size on the strength can be obtained from Equation 1.

$$\left(\frac{\sigma_2}{\sigma_1}\right) = \left(\frac{V_{e1}}{V_{e2}}\right)^{1/m} \text{ or } = \left(\frac{S_{e1}}{S_{e2}}\right)^{1/m} \quad (1)$$

where  $\sigma$  is the mean strength,  $m$  is the Weibull modulus,  $V_e$  is the effective volume,  $S_e$  is the effective surface (prismatic), and 1 and 2 represent the different specimen geometries.

If volume flaws dominate, then the effective volume ratio is used on the right-hand side of the Equation 1. If surface flaws control the strength, then the effective surface ratio is used.  $V_e$  and  $S_e$  must be calculated according to Equations 2 through 5.

**For three-point flexure:**  $V_e = V/[2(m+1)^2]$  (2)  
(center-point loading)

$$S_e = [L/(m+1)] [\{H/(m+1)\} + B] \quad (3)$$

**For four-point flexure:**  $V_e = V(m+2)/[4(m+1)^2]$  (4)  
(1/4-point loading)

$$S_e = [L(m+2)/2(m+1)] [\{H/(m+1)\} + B] \quad (5)$$

where:  $L$  = length of the outer span,  $H$  = specimen height,  $B$  = specimen base, and  $V$  = volume of the material tested within the outer span.

From Reference 9 the manufacturer's strength was determined with a three-point flexure configuration of 3 mm x 4 mm x 30 mm. The Weibull modulus value used in these calculations is 6, which was obtained from the room temperature strengths determined in this study. For either flaw type the four-point flexure strength should be  $\approx 24\%$  lower than the three-point value. This is an excellent agreement with the actual value. It does not, however, allow for the determination of which flaw type controls the strength.

The difference between the as-received strength and the strength after heat treatment is due to a change in the surface compressive stresses which are introduced during machining. Studies<sup>18-22</sup> have shown that machining or grinding of partially stabilized zirconia causes the tetragonal-to-monoclinic transformation which introduces a compressive stress on the surface. These stresses can be quite large, as shown by Green, et al.,<sup>22</sup> who measured residual stresses of  $\approx -1$  GPa on the surface of a 40%  $Al_2O_3$ /60%  $ZrO_2$  composite. Preliminary analysis to determine the residual stresses in this material was done by applying the  $\sin^2 \psi$  technique to X-ray diffraction results that were obtained with  $CrK\alpha$  radiation. The stresses ranged from -123 MPa to -318 MPa for the as-received condition and decreased to between -83 MPa and -218 MPa after heat treatment. At elevated temperatures thermal relaxation of these stresses is expected but studies<sup>23,24</sup> have shown that it is a time-dependent process even at temperatures as high as 1600°C. The time-dependent aspect is attributed to microcrack healing that must occur before all of the m- $ZrO_2$  is retransformed and the stresses are relieved.<sup>23</sup>

X-ray diffraction results provide further support for the relaxation of the stresses and retransformation of the  $ZrO_2$ . The results showed the monoclinic  $ZrO_2$  content on the surface to be  $\approx 9\%$  in the as-received case, but after heat treatment there was no detectable monoclinic  $ZrO_2$ . This indicates that a sufficient amount of stress was relieved to enable the monoclinic  $ZrO_2$  to transform to the tetragonal phase.

Fractography revealed that for all specimens (in both conditions) the strength-limiting defect was a crack created during machining. The only difference between the two conditions was the location of the defect in the specimen. In the as-received case, the defects are located at the edge/chamfer (see Figure 1), but for the heat treatment specimens the defects are located on the tensile surface, as shown in Figure 2. The reason for this difference in flaw location is unknown.



Figure 1. Fracture surface of an as-received specimen. The arrowheads indicate the machining damage on the chamfer. The small arrows outline the semi-elliptical crack created by the machining. Fracture stress = 1732 MPa. "T" denotes tensile surface.

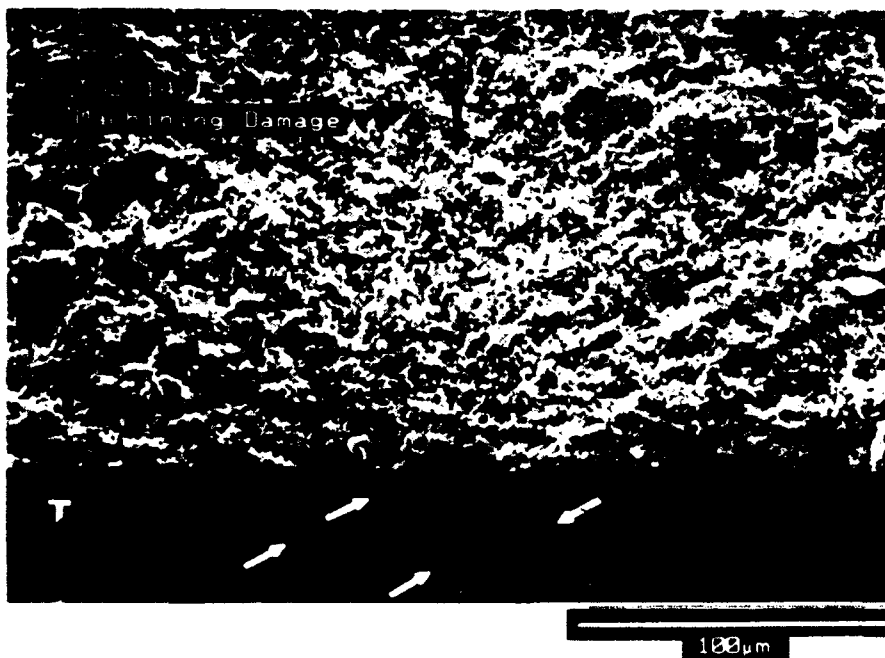


Figure 2. Fracture surface of a specimen after 100 hours at 1000°C. White arrows indicate the machining damage on the tensile surface. Black arrow heads outline the semi-elliptical cracks that were created. Large black arrows outline the mirror region. Fracture stress = 1552 MPa. "T" denotes tensile surface.

The average grain size of the zirconia and alumina, in the as-received conditions, was  $0.4\ \mu\text{m}$  and  $0.6\ \mu\text{m}$ , respectively. No grain growth occurred for either material as a result of the heat treatment. Figure 3 is an example of the microstructure of this material.

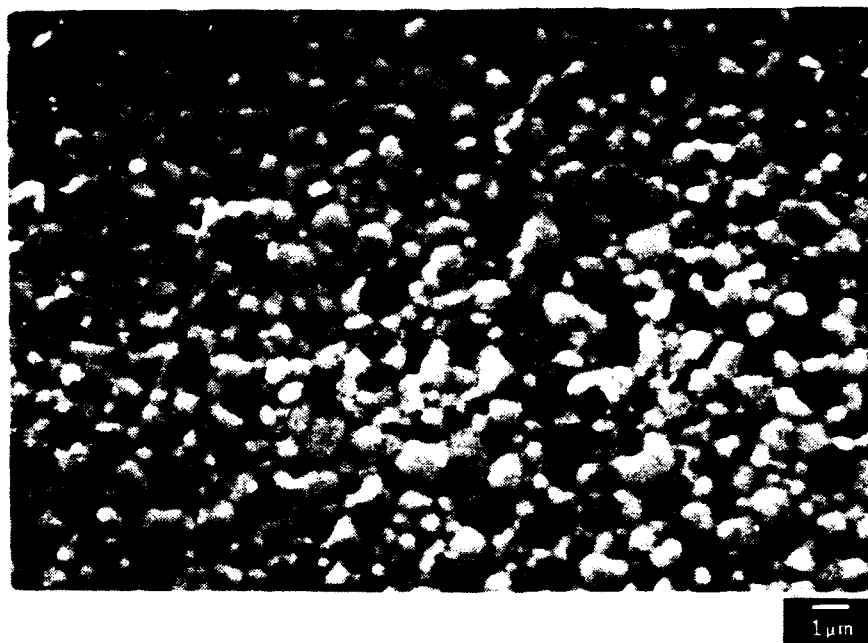


Figure 3. Micrograph of the microstructure. Black grains are alumina and the grey grains are zirconia.

A comparison of these properties with those of the Y-TZPs previously examined<sup>1</sup> shows that this TZP/ $\text{Al}_2\text{O}_3$  is significantly stronger but its toughness is only average. The strongest monolithic Y-TZP in Reference 1 is HIPed and has strength of 1261 MPa ( $K_{\text{IC}} = 5\ \text{MPa}\sqrt{\text{m}}$  and grain size =  $0.4\ \mu\text{m}$ ) while the toughest was sintered and has a  $K_{\text{IC}} = 8.5\ \text{MPa}\sqrt{\text{m}}$  ( $\sigma = 633\ \text{MPa}$  and grain size =  $0.6\ \mu\text{m}$ ). The high strength and average toughness for this composite is not surprising since fine grains, HIPing, and the addition of  $\text{Al}_2\text{O}_3$  have all been shown to lead to high strength values. The low toughness is an indication that the  $\text{ZrO}_2$  grains are well below the critical size necessary to maintain the tetragonal phase, and the critical stress necessary for transformation is high.

#### Static Fatigue Life

The static fatigue life between  $800^\circ\text{C}$  and  $1200^\circ\text{C}$  was determined before and after heat treatment (see Figure 4). In both cases the static fatigue life decreases as the temperature increases. This is not unexpected since as the temperature approaches  $1100^\circ\text{C}$  (the approximate temperature at which the tetragonal phase is stable), the driving force for the tetragonal-to-monoclinic transformation gradually decreases.

Between  $800^\circ\text{C}$  and  $1000^\circ\text{C}$  the material in the as-received condition performs slightly better. This may be due to the compressive stresses created in the surface during machining. As stated previously, thermal relaxation of these stresses is a time-dependent process<sup>23</sup> and, in this study, exposure times are modest. Above  $1000^\circ\text{C}$  the performance is essentially the same for both

conditions. Because the tetragonal phase is becoming increasingly stable as the temperature increases, the strength at temperature becomes dependent upon the microstructural parameters such as grain size and grain size distribution. Since these parameters remain constant even after heat treatment the performance is expected to be similar.

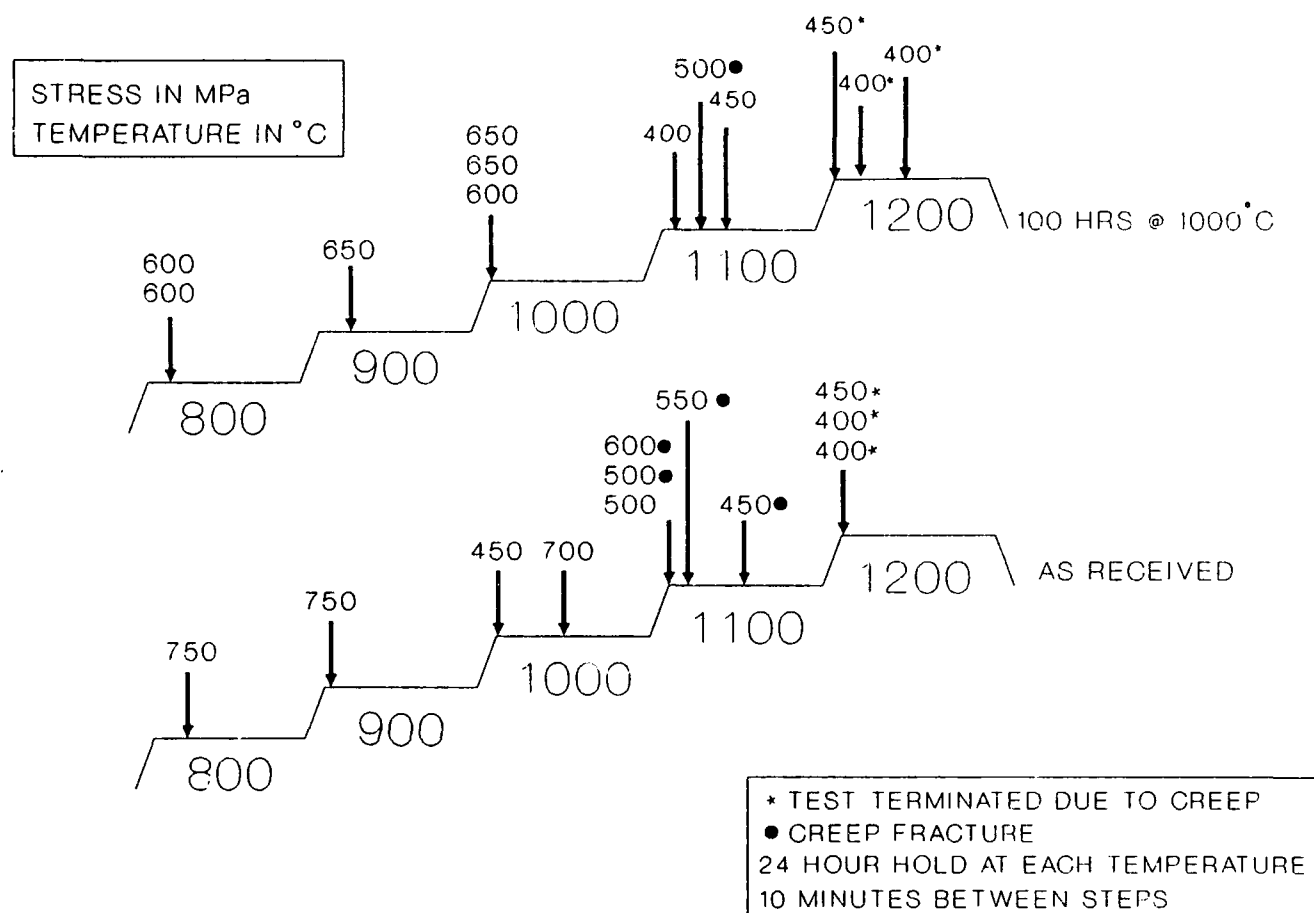


Figure 4. STSR results before heat treatment (as-received) and after exposure to 1000°C for 1000 hours.

Fractographic analysis indicates that during the first three steps of this test fracture initiates at damage created during machining (see Figure 5). Starting at 1100°C the failure mechanism changed to slow crack growth which is presumed to have started from the machining damage, as seen in Figures 6 and 7. This is the case for both conditions. To the author's knowledge, this is the first instance where slow crack growth has been reported in zirconia-based ceramics. Permanent strain was measured on specimens which survived into the 1100°C and 1200°C steps. This value ranged from a low of 0.27% at 1100°C to a high of 4.7% at 1200°C. No permanent strain was noticeable for either condition below 1100°C.

Several of the Y-TZPs tested in Reference 4 have similar ZrO<sub>2</sub> grain sizes, but the two- to three-fold improvement in the static fatigue life of this material appears to be the result of the very small flaws and the addition of the Al<sub>2</sub>O<sub>3</sub>. The best Y-TZP tested in Reference 4 failed from large porosity related flaws. Plastic deformation was evident at temperatures above 1100°C but no slow crack growth was observed.



Figure 5. Fracture surface of an STSR specimen which failed after 0.1 hours at 1000°C under 650 MPa applied stress. Surface machining damage is noted by the arrowhead and the semi-elliptical crack by the small white arrow. Large white arrows outline the mirror region. "T" denotes the tensile surface.

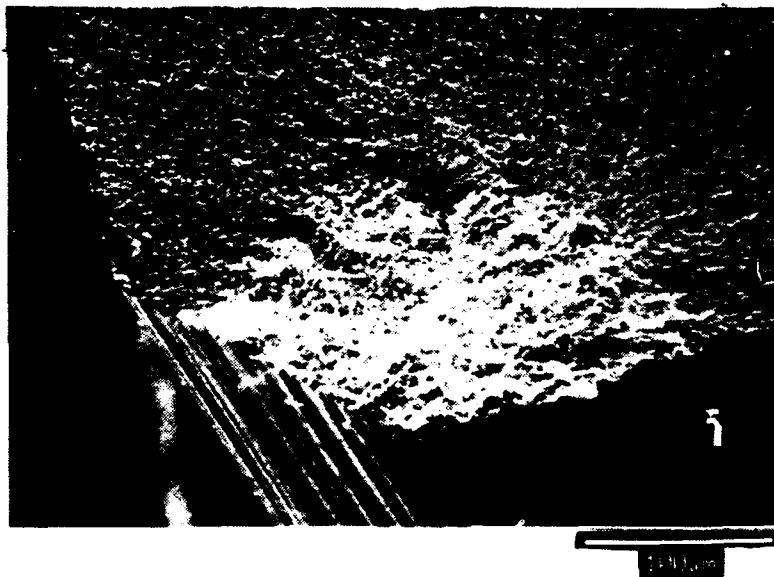


Figure 6. Slow crack growth region on an as-received specimen which failed after 0.3 hours at 1200°C under a 450 MPa applied stress. "T" denotes the tensile surface.

Although the static fatigue life of this material is markedly better than the Y-TZPs previously tested,<sup>2,4</sup> it is still below the strength-at-temperature requirements (>800 MPa at >982°C)<sup>25</sup> for advanced engines.

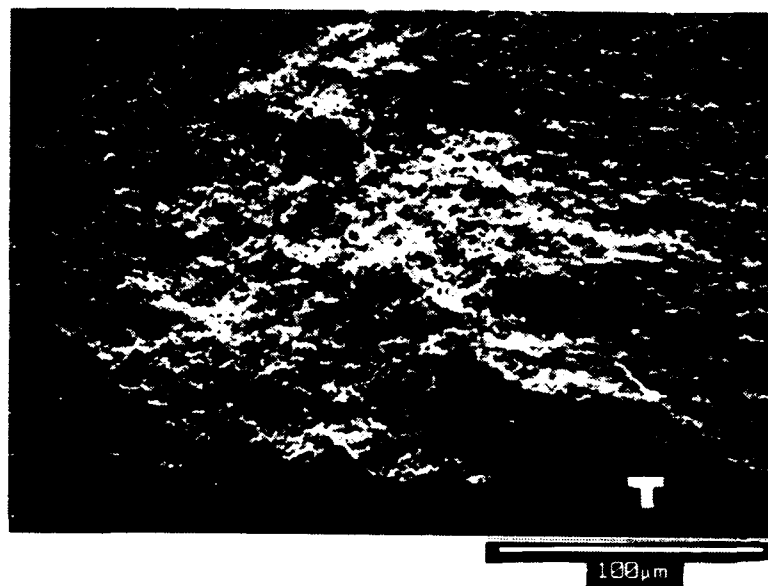


Figure 7. Slow crack growth region on a specimen which failed after six hours at 1100°C under a 500 MPa applied stress. Specimen was heat treated for 100 hours at 1000°C before STSR testing. "T" denotes the tensile surface.

### CONCLUSIONS

The room temperature flexure strength of a TZP/Al<sub>2</sub>O<sub>3</sub> ceramic was determined to be 1838 MPa with a strength decrease of 18% after 100 hours at 1000°C. Even with this strength decrease it was still the strongest zirconia ceramic tested by this laboratory. Room temperature fracture toughness was determined to be  $\approx 5$  MPa $\sqrt{\text{m}}$  before and after the heat treatment. This value is about average for a transformation toughened zirconia.

The static fatigue life between 800°C and 1200°C was also examined in this study. At temperatures below 1100°C fracture was controlled by machining damage. At 1100°C and higher slow crack growth became the dominant failure mechanism. The behavior was significantly better than monolithic Y-TZP materials but it still fell short of the strength-at-temperature requirement for use in advanced engines.

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## REFERENCES

1. SWAB, J. J. *Properties of Ytria-Tetragonal Zirconia Polycrystal (Y-TZP) Materials After Long-Term Exposure to Elevated Temperatures*. U.S. Army Materials Technology Laboratory, MTL TR 89-21, prepared for Oak Ridge National Laboratory for the U.S. Department of Energy under Interagency Agreement DE-AI05-84OR21411, March 1989.
2. SCHIOLER, L. J. *Effect of Time and Temperature on Transformation Toughened Zirconias*. U.S. Army Materials Technology Laboratory, MTL TR 87-29, prepared for Oak Ridge National Laboratory for the U.S. Department of Energy under Interagency Agreement DE-AI05-84OR21411, June 1987.
3. LARSEN, D. C., and ADAMS, J. W. *Long-Term Stability and Properties of Zirconia Ceramics for Heavy Duty Diesel Engine Components*. Prepared for NASA-Lewis Research Center for U.S. Department of Energy under Contract DEN 3-305 NASA CR-174943, September 1985.
4. SWAB, J. J. *Performance of Y-TZP Materials Between 800°C and 1200°C*. U.S. Army Materials Technology Laboratory, MTL TR 90-3, prepared for Oak Ridge National Laboratory for the U.S. Department of Energy under Interagency Agreement DE-AI05-84OR21411, January 1990.
5. SWAB, J. J. *Low Temperature Degradation of Y-TZP Materials*. U.S. Army Materials Technology Laboratory, MTL TR 90-4, prepared for Oak Ridge National Laboratory for the U.S. Department of Energy under Interagency Agreement DE-AI05-84OR21411, January 1990.
6. WATANABE, M., IIO, S., and FUKUURA, I. *Aging Behavior of Y-TZP*. *Advances in Ceramics, Science, and Technology of Zirconia II*. N. Claussen, M. Rühle, and A. H. Heuer, eds., The American Ceramic Society, Columbus, OH, v. 12, 1984, p. 391-398.
7. LU, H. Y., and CHEN, S. Y. *Low Temperature Aging of t-ZrO<sub>2</sub> Polycrystals with 3 mole% Y<sub>2</sub>O<sub>3</sub>*. *J. Am. Ceram. Soc.*, v. 70, no. 8, 1987, p. 537-541.
8. SATO, T., OHTAKI, S., ENDO, T., and SHIMADA, M. *Humidity Dependence of Phase Transformation from Tetragonal to Monoclinic Structure in Ytria-Doped Tetragonal Zirconia*. *High Tech. Ceramics*. P. Vincenzini, ed., Elsevier Science Publishers, B.V., Amsterdam, The Netherlands, 1987, p. 281-288.
9. TSUKUMA, K., UEDA, K., and SHIMADA, M. *Strength and Fracture Toughness of Isostatically Hot-Pressed Composites of Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub> - Partially-Stabilized ZrO<sub>2</sub>*. *J. Am. Ceram. Soc.*, v. 68, no. 1, c-4-c-5, 1985.
10. TSUKUMA, K., and TAKAHATA, T. *Mechanical Property and Microstructure of TZP and TZP/Al<sub>2</sub>O<sub>3</sub> Composites*. *Advanced Structural Ceramics*. P. F. Becher, M. V. Swain, and S. Sömiya, eds., Materials Research Society Symposia Proceedings, v. 78, 1987, p. 123-136.
11. TSUKUMA, K., TAKAHATA, T., and SHIOMI, M. *Strength and Fracture Toughness of Y-TZP, Ce-TZP, Y-TZP/Al<sub>2</sub>O<sub>3</sub>, and Ce-TZP/Al<sub>2</sub>O<sub>3</sub>*. *Advances in Ceramics, Science and Technology of Zirconia III*. S. Sömiya, N. Yamamoto, and H. Hanagida, eds., The American Ceramic Society, Columbus, OH, v. 24, 1988, p. 721-728.
12. LANGE, F. F. *Transformation Toughening-Part 3: Experimental Observations in the ZrO<sub>2</sub>-Y<sub>2</sub>O<sub>3</sub> System*. *J. Mat. Sci.*, v. 17, 1982, p. 240-246.
13. QUINN, G. D., SWAB, J. J., and SLAVIN, M. J. *A Proposed Standard Practice for Fractographic Analysis of Monolithic Advanced Ceramics*. *Fractography of Glasses and Ceramics II*, Ceramic Transactions, v. 17. V. Frechette and J. Varner, eds., American Ceramic Society, Westerville, OH, 1991, p. 309-361.
14. CHANTIKUL, P., ANTIS, G. R., LAWN, B. R., and MARSHALL, D. B. *A Critical Evaluation of Indentation Techniques for Measuring Fracture Toughness: II, Strength Method*. *J. Am. Ceram. Soc.*, v. 64, no. 9, 1981, p. 539-543.
15. QUINN, G. D., and KATZ, R. N. *Stepped Temperature Stress Rupture Testing of Silicon-Based Ceramics*. *Am. Ceram. Soc. Bull.*, v. 57, no. 11, 1978, p. 1057-1058.
16. MENDELSON, M. I. *Average Grain Size in Polycrystalline Ceramics*. *J. Am. Ceram. Soc.*, v. 52, no. 8, 1969, p. 443-446.
17. WEIL, N. A., and DANIEL, I. M. *Analysis of Fracture Probabilities in Nonuniformly Stressed Brittle Materials*. *J. Am. Ceram. Soc.*, v. 47, no. 6, 1964, p. 268-274.
18. GARVIE, R. C., HANNINK, R. H. J., and PASCOE, R. T. *Ceramic Steel*. *Nature*, v. 258, 1975, p. 703-704.
19. GUPTA, T. K. *Strengthening by Surface Damage in Metastable Tetragonal Zirconia*. *J. Am. Ceram. Soc.*, v. 63, no. 1-2, 1980, p. 117.
20. CLAUSSEN, N. and PETZOW, G. *Strengthening and Toughening Models in Ceramics Based on ZrO<sub>2</sub> Inclusions* in *Energy and Ceramics*, P. Vincenzini, ed., Elsevier Press, Amsterdam, 1980, p. 680.
21. SWAIN, M. V. *Grinding-Induced Tempering of Ceramics Containing Metastable Tetragonal Zirconia*. *J. Mat. Sci. Lett.*, v. 15, 1980, p. 1577-1579.
22. GREEN, D. J., LANGE, F. F., and JAMES, M. R. *Factors Influencing Residual Surface Stresses Due to a Stress-Induced Phase Transformation*. *J. Am. Ceram. Soc.*, v. 66, no. 9, 1983, p. 623-629.
23. GREEN, D. J. *Critical Microstructures for Microcracking in Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> Composites*. *J. Am. Ceram. Soc.*, v. 65, no. 12, 1982, p. 610-614.
24. GREEN, D. J., LANGE, F. F., and JAMES, M. R. *Residual Surface Stresses in Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> Composites*. *Advances in Ceramics, Science and Technology of Zirconia II*. N. Claussen, M. Rühle, and A. H. Heuer, eds., The American Ceramic Society, Columbus, OH, v. 12, 1984, p. 240-250.
25. BRYZIK, W., and KAMO, R. *TACOM/Cummins Adiabatic Engine Program*. SAE Paper 830314 in SP-543 - The Adiabatic Diesel Engine, SAE International Congress and Exposition, Detroit, MI, 1983, p. 21-45.



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PROPERTIES OF A TZP/Al<sub>2</sub>O<sub>3</sub> COMPOSITE AFTER  
LONG-TERM EXPOSURE AT 1000°C.  
Jeffery J. Swab and Steven C. Stowell

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A commercially available tetragonal zirconia polycrystal ceramic containing 20 w/o  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> was examined for potential use in advanced engines. Room temperature strength and toughness were determined, as well as the static fatigue life between 800°C and 1200°C. All of these properties were determined before and after a 100 hour exposure at 1000°C. The room temperature flexure strength was 1838 MPa before exposure and it decreased 23% after exposure. The toughness was  $\sim 5$  MPa $\sqrt{m}$  and did not change with the exposure. Static fatigue testing revealed that fracture below 1100°C was due to the cracks created during machining. At 1100°C and higher slow crack growth became the dominant failure mechanism. Overall, the high temperature static fatigue life is better than yttria-tetragonal zirconia polycrystal materials without Al<sub>2</sub>O<sub>3</sub> but it still does not meet the strength-at-temperature requirements (>800 MPa at 982°C) for use in advanced engines.

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